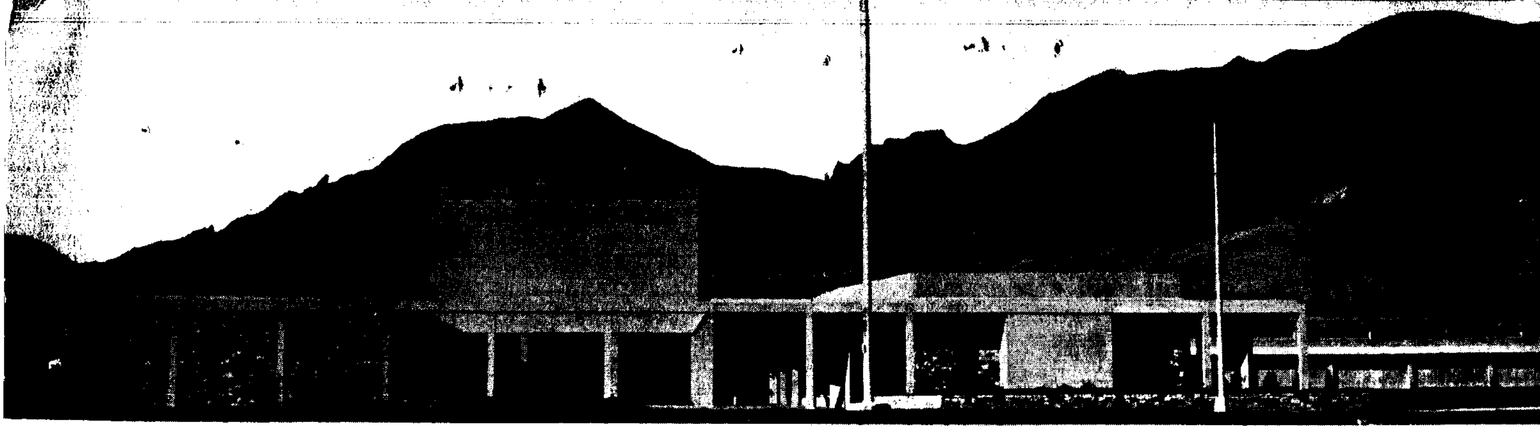

NBS REPORT

9275

TWENTY-FIFTH PROGRESS REPORT

R-45 to

National Aeronautics and Space Administration

on

Cryogenic Research and Development

for

Period Ending March 31, 1967

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
BOULDER LABORATORIES
Boulder, Colorado

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NATIONAL BUREAU OF STANDARDS REPORT

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31502-3150420	March 31, 1967	9275
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Cryogenics Division
Institute for Materials Research
National Bureau of Standards
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U.S. DEPARTMENT OF COMMERCE

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Preliminary Planning									
Not Started									
Active Coord. with Lewis, MSFC, LASL							X	X	X

* continuing effort

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Data Taking								X	
Apparatus Construction									X
Apparatus Designing									
Preliminary Planning							X		
Not Started									
Active Coord. with Lewis, MSFC, LASL		X	X	X	X	X			

* continuing effort

1. Physical Properties of Cryogenic Fluids

1.0 General Comments

Personnel contributing during this period were D. E. Diller, W. J. Hall, M. C. Jones, H. M. Roder, and B. A. Younglove.

1.1 Dielectric Constant of Solid Parahydrogen

Approximately 30 points have been taken of the dielectric constant of solid hydrogen on the melting curve. These measurements combined with densities reported by Dwyer and Cook (J. Chem. Phys. 43, 805 (1965)) but modified below 17°K as shown below, are used to calculate the polarizibility (Clausius-Massotti function) of the solid.

These measurements cover the density range 0.0869 to 0.0945 gm/cm^3, corresponding to the temperature range 14.2 to 20.4°K. Measurements to 22.6°K will be completed shortly and will complete the data for the solid dielectric constant.

The polarizibilities are almost independent of density to the precision of the measurement (within 0.1%). A good representation is

$$P = 1.0100 - 0.0364 \rho \; cm^3/gm,$$

with ρ in gm/cm^3. Extrapolation of Stewart's equation (J. Chem. Phys. 40, 3297 (1964)) to the solid densities gives numbers too low, by about 0.34%, and for that matter does not fit new data of this laboratory on the liquid for densities exceeding that reported by Stewart, the polarizibilities in this case being about 0.14% higher than the measured value at 0.089 gm/cm^3. However, very good agreement was found for the 16 data points taken in the same density range as reported by Stewart.

About 18 points were taken in the compressed liquid region above 0.080 gm/cm^3 (the maximum in Stewart's paper). Some of these are at densities equivalent to those of the solid. It is seen that the solid polarizibilities exceed that of the liquid by about 0.4 to 0.5%, the liquid being less.

Values for the dielectric constant of solid parahydrogen can be calculated from the above equation for polarization together with the equation for the density of solid parahydrogen on the melting curve

$$\rho = 0.069198 + 0.001232\ T\ \mathrm{gm/cm^3},$$

with T in °K, by using the Clausius Mossotti relation

$$P = \frac{\epsilon - 1}{\epsilon + 2}\ 1/\rho\ .$$

The above equation for density should be used in preference to equation (3) of Dwyer as it is more accurate below 17°K as a result of the melting pressures and temperatures recently measured on this project. The above equation gives densities about 0.24% lower near the triple point; it fits the adjusted data below 17°K as well as Dwyer's data above 17°K within the experimental precision. It is interesting to note that the data are now best represented by an equation linear in density rather than one linear in specific volume, as previously used.

1.2 Refractive Index Measurements on Hydrogen Gas

Refractive index measurements on parahydrogen at 35°K are in progress. The initial measurements at this temperature gave a peculiar density dependence for the Lorentz-Lorenz Function. Vapor pressure measurements on parahydrogen at 30° - 32°K showed the difficulty to be a thermometry error. The thermometer was fixed by washing the glass surface and platinum leads with alcohol. Subsequent vapor pressure measurements agreed with previous measurements in the P-V-T apparatus to 1 part in 10,000. The thermometry error should not affect our previous refractive index-density measurements at higher temperatures by more than one part in 10,000.

The density dependence of the Lorentz-Lorenz Function at 35°K is similar to the density dependence at 100°K. The low density intercept appears to be independent of temperature in this temperature range. It is not likely that refractive index measurements can be obtained much closer than 2 degrees from the critical temperature with this interferometer because the contrast of the interference pattern becomes quite poor when passing through the critical density.

1.3 TAB Code and Tables of Evaluated Data

An error analysis is required for a final publication describing the series of TAB Code programs. The task has been divided into two parts -- analysis of the transport properties, and analysis of errors in the PVT surface and the related or derived properties.

The analysis incorporates a survey of the PVT data from which will be compiled a set of critical tables for the task on tables of evaluated data. Above 100°K a single PVT surface should suffice because the differences in PVT between normal and parahydrogen are no greater than the variation between different experimenters. The next step then is to find the "best" surface for the PVT data above 100°K.

For the TAB programs there are three contributions to the total error:

- ϵ_1 the difference between the source used and a "best" source,
- ϵ_2 the uncertainty in the coefficients of the equation of state used,
- ϵ_3 the error caused by linear interpolation in a fixed grid of points.

Explicit values may be given for ϵ_3 if the integrals formed from the equation of state and required for the derived functions can be handled in closed form. This would be true for an equation such as the BWR but not for Woolley's equation. Specifically, one can calculate the uncertainty in enthalpy caused by the deviations between experimental PVT data and the (fitted) PVT surface.

If it is desired to find the error ϵ_3, it becomes necessary to reprogram all of the various sources used; this is not practical. A shortcut would be to compare the TAB Code programs and the "best" representation. The deviations so obtained provide the errors ϵ_1 and ϵ_3 lumped together. Even a reasonable approximation to the "best" representation yields an acceptable estimate of the error pattern. While it may appear that the interpolation error ϵ_3 will by far outweigh the error ϵ_1, the latter should not be neglected. As an example, take an enthalpy comparison between the source (AiResearch) and a "best" value (Woolley).

At 1000°R and 14.696 psia, we have 3409 vs 3409 BTU/#.

At 1000°R and 3000 psia, we have 3474 vs 3477 BTU/#.

The difference is 3 BTU/#, but this difference is equivalent to a change of 125 psia in pressure.

1.4 Absorption of Thermal Radiation in Liquid Hydrogen

Experimental work has commenced on the measurement of absorption of radiation of thermal wavelengths in liquid hydrogen. A cell was constructed of brass with crystal quartz windows allowing the passage of a spectrophotometer beam. The spacing of the windows is adjustable by the insertion of a brass spacer.

The useable wavelength region of the cell is determined by the window material and, in the case of crystal quartz, is for wavelengths greater than about 50 microns and less than 5. To date the cell has been tested with liquid nitrogen condensed in it. The pure rotational spectrum of nitrogen was obtained between 500 and 50 microns with a 1/2-inch window spacing.

Preliminary observations with liquid normal hydrogen show that, with this path length in the same wavelength range, absorption is very weak; in general it is less than 10%. It is therefore proposed to repeat observations with an increased path length.

2. Cryogenic Properties of Solids

2.1 Thermal Conductivity of Solids

2.1.1 General Comments

The objective of this project is to determine the thermal conductivities of several aerospace alloys and standard reference materials from liquid helium temperatures to above 120°K.

Personnel contributing during the present reporting period are J. G. Hust and R. L. Powell.

2.1.2 Program Status

During the current reporting period the Ti + 5Aℓ + 2.5 Sn sample was placed in the apparatus. The apparatus was checked for leaks at room and liquid nitrogen temperatures. Preliminary tests on the completed assembly were carried out and measurements above liquid nitrogen were begun. Data were taken at temperatures between 75°K and 145°K. The electrical resistivity and thermoelectric power data are believed accurate to 1% or better. The present uncertainty in thermal conductivity data is approximately 20% and will be improved significantly during the next reporting period. This high uncertainty is caused by:

(a) the very low thermal conductance of this sample,

(b) inability to match the shell temperature to the sample temperature,

(c) temperature drift of the floating sink and the bath temperature,

(d) thermocouple calibration uncertainty.

The low thermal conductance of this sample allows for large relative heat leaks along the connecting wires to the shell. Since the shell has always been hotter than the sample at the middle, the problem is further exaggerated. Temperature drift of the sample introduces appreciable error into these measurements because of the relatively large heat capacity of the sample heater and thermocouple holders as compared to the heat capacity of the sample. With such a poor conductor, the energy going into enthalpy changes as temperature drifts occur is appreciable compared to the energy flowing through the sample; thus the energy generated by the sample heater is not the energy flowing through the sample at all points. The results based on these measurements are calculated with rough estimates of the sensitivity of the AuFe vs. Chromel thermocouples. Preliminary measurements of the sensitivity of these thermocouples shows these rough estimates to be significantly in error.

During the next reporting period, changes will be incorporated into the apparatus to reduce the large descrepancies described above. Also the sensitivities of the thermocouples will be more accurately determined. The sample cross-sectional area for the Ti A-110, Inconel 718 and Hastalloy-X will be increased to 1 cm^2. A new sample heater will be made to reduce its heat capacity. These changes will reduce the effects of temperature drift and of sample-to-shell temperature differences. Copper wires will be connected between the floating sink and the shell to introduce refrigeration to the shell in the areas where it has been too hot. The hot shell exists because the thermal conductivity of the shell material (stainless steel) increases relatively more rapidly with temperature than the sample. Before the apparatus is modified, a run will be made at liquid helium temperature to determine if any unforeseen difficulties exist at very low temperatures.

During the current reporting period, we have also assisted the General Dynamics/Fort Worth thermal conductivity radiation effects program. General Dynamics personnel were given a demonstration of NBS data-taking methods. At another meeting at Boulder, personnel from General Dynamics and Aerojet-General were informed of problems regarding sample size, temperature drift, and energy losses from a sample. Data analysis was also discussed and a method of data correlation of several runs was presented. The importance of proper data spacing and sequencing was stressed, and a specific method was outlined.

During the next reporting period, the measurements on Ti A-110 should be completed, and data analysis will be in progress.

2.2 Thermocouple Thermometry

2.2.1 General Comments

The thermocouple calibration apparatus is operational. The measurement, temperature control, and insulating vacuum systems have been tested at liquid nitrogen temperatures. Initial checkout data were taken in February. At present, precise spot checks are being made above 100°K to determine the degree of stability in the temperature control system and to obtain preliminary data necessary for the thermal conductivity project.

Personnel contributing during this period were L. L. Sparks and R. L. Powell.

2.2.2 Program Status

Proper measurement sequences and the number of necessary measurements at each temperature have been tentatively determined. The measurement plan is being tested in our preliminary runs. Figure 1 is a schematic of our measurement graph. Voltages will be read between pairs of wires as indicated by the lines when the two junctions are being maintained at temperatures T_1 and T_2. The triple lines indicate a measurement of major importance, the double lines a measurement of importance, and the single line a measurement of minor importance. This design yields the maximum information from a minimum number of measurements. These measurements allow statistically sound limits to be derived for the final results.

Spot calibrations have been made of the Chromel vs. gold-iron thermocouple pair being used in the thermal conductivity experiment. Until these spot calibrations were done, there were no reliable data on the thermoelectric behavior of the gold-iron above 100°K. Preliminary measurements also have been made on all of the combinations of wires shown in Figure 1.

Two problems developed during the early liquid nitrogen tests, and corrective measures have been taken. First, the cryostat vacuum seal cracked when immersed in liquid nitrogen. We found that the stainless steel cryostat was slightly out-of-round which caused differential contraction in the solder seal. Second, there was excessive signal noise in certain thermocouple pairs. This noise was traced to extension wires which acted as magnetic pick-up coils.

Various functional forms are being considered as means of representing our experimental data. A detailed system error analysis is about to be started and will aid in our selection of the best curve fitting procedure.

The computer program to be used to analyze experimental data has been blocked out, but not written in detail. Upon selection of a functional form to represent the data, it will be completed in detail and checked using preliminary results.

The experimental measurements for this particular set of thermocouple wires will have been completed by the end of the next report period. The associated data reduction and reporting should also be started.

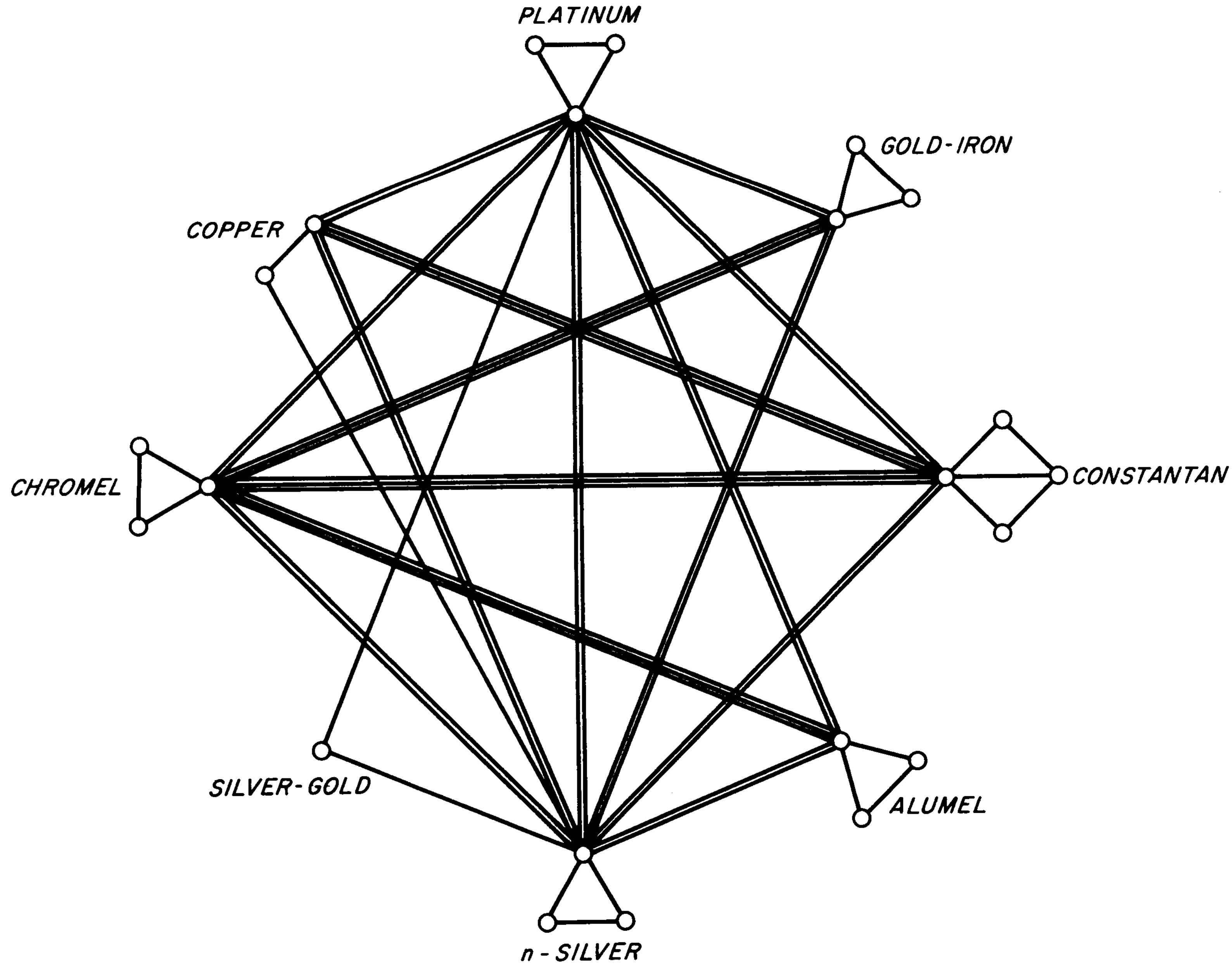

Fig. 1 Graph of Thermocouple Measurement System

A. 6169

2.2.3 Summary

The entire apparatus is in operation, and preliminary results have been taken. Spot calibrations have been made for thermal conductivity program. The remaining tasks are to complete the error analysis, to select a method of functionally representing our experimental data, to write the details of the computer program, to take the experimental data, and finally to develop suitable tables of data.

2.3 Thermal Expansion

2.3.1 General Comments

The objective of this project is to measure the thermal expansion coefficients of several aerospace alloys and standard reference materials from liquid hydrogen temperature, 20°K, to room temperature, 293°K. Materials to be measured are determined as those needed for cryogenic design in the aerospace industry and for the reduction of electrical resistivity measurements of standard reference materials.

The measurement method and apparatus will be that of Arp, et al.[1], previously used at this laboratory. An 8-inch long sample is slowly cooled by helium exchange gas after the apparatus is immersed in a cryostat containing liquid nitrogen or liquid hydrogen. The differential contraction is transmitted to a dial gauge at room temperature by concentric quartz tubes. Temperature is determined by thermocouples attached to the sample.

The data will be compiled in a standard tabular form[2] showing both the expansion referred to room temperature, $(L_{293} - L_T)/L_{293}$, and the expansion coefficient, $(1/L_{293})(dL/dT)$.

---

[1] V. Arp, J. H. Wilson, L. Winrich, and P. Sikora, Cryogenics 2, 230 (June 1962).

[2] R. J. Corruccini and J. J. Gniewek, NBS Monograph 29, May 1961.

Personnel contributing during this period were A. F. Clark and G. Rogacki.

Additional references (and data) were found for the following materials which will supplement the data referenced in the NERVA Data Book:

Aluminum 2219

Inconel X

Iron Alloys

347

660 (A286)

These data are being compiled and will be tabulated in the next quarterly report. A contact was made with Mr. S. Goodman of Aerojet-General Corporation, Sacramento, California in order to make him aware of our planned scope of work and in order to obtain maximum coordination. The next quarterly report (June) will precede the next supplement to the NERVA Data Book (August).

The dilatometer apparatus used by Arp, et al., was removed from storage and evaluated for necessary changes and replacements. The only significant change will be to automate the data acquisition with the use of a differential transformer to electronically measure the expansion. The thermal expansion of OFHC copper[1,2] will be used to recalibrate the apparatus. Copper for this sample has been obtained as well as Aluminum 5083, Hastalloy X, and S.S. 633 (AM350) for initial measurements.

In the next reporting period, a literature search for standard reference materials and a compilation of existing data will be completed, the apparatus will be made operational and calibrated, and the necessary samples will be acquired. It is anticipated that initial measurements will have begun.

2.3.2 Program Status

A literature search for data on the aerospace alloys based upon the materials in the NERVA Materials Properties Data Book[3] has been completed. Data on the thermal expansion for the temperature range 20 - 293°K was found for the following materials not complete in the NERVA Data Book:

Aluminum Alloys

356

2014

5456

7075

7079

Titanium Alloys

Ti-5Al-2.5Sn (A-110 AT)

Ti-6Al-4V (C-120 AV)

Molybdenum

Tantalum

Tungsten

Copper OFHC

Iron Alloys

4340

301

303

316

630 (17-4PH)

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[3] Materials Properties Data Book, Aerojet-General Report 2275, 30 Sept. 1966.

3. Consultation and Advisory Services

3.0 General Comments

Consultation and advisory services in the general field of cryogenic engineering have continued in several NASA program areas: Centaur (funded separately) and NERVA.

3.1 Centaur Program - Robert W. Arnett

Contact with NASA-LeRC personnel has been frequent. One trip was made to Cape Kennedy by R. W. Arnett and D. B. Chelton, and one trip to San Diego was made by R. W. Arnett and R. O. Voth.

3.1.1 Stratification and Pressurization

The results obtained from the computer program covered a wide range of heat flux parameters, both in intensity and location. Pressure rise rates resulting from these inputs qualitatively have the type of time variation experienced during ground tests. However, quantitatively the pressure rise rates are higher than test values by a factor of two. A study of the equations is underway to determine if certain of the assumptions made in the development are not wholly justified. In particular, expressions involving the decay of the boundary layer are being scrutinized.

Due to this unforeseen difficulty, the printing and distribution of NBS Report 9266, "Mathematical Analysis of Thermal Stratification and Self Pressurization in a Closed Container," has been delayed.

A presentation of the mathematical development and program results was made at San Diego, GD/C, on March 6 by R. W. Arnett.

3.1.2 Flight Data Analysis

Reduction of flight data tapes of the AC-9 launch to graphical form has been completed. Analysis of these graphs is progressing and it is expected that completion of this phase will be accomplished within the next month.

3.1.3 Helium Facility Study

R. W. Arnett and D. B. Chelton traveled to Kennedy Space Center on January 23-27 to observe the operation of the helium liquefier at Pad 36B. As a result of the observations made there together with a study of the system, an informal report has been prepared and submitted to the Centaur project office.

3.2 NERVA Program - Alan F. Schmidt, Daniel H. Weitzel, Jesse Hord

Throughout the quarter, the following information was supplied to various organizations at the request of SNPO-C:

Physical properties data on methane and ammonia, and cryogenic and space environmental data on selected plastics [for WANL via SNPO-C].

NBS hydrogen properties TAB Code decks, T-S charts, reports, and reprints [AGC].

A bibliography on the properties of propane, and selected data [NASA-Lewis].

Discussions concerning flight vehicle cryogenic temperature measurement systems, cooldown time for cryogenic pipelines, and uncertainties in the generally accepted values of thermal conductivity of fluid para-hydrogen [SNPO-C].

The NERVA Instrumentation Development Design Review Meeting was attended at AGC/REON in Sacramento, California on March 7-8; a classified trip report with NBS comments on this business was prepared and forwarded to SNPO-C.

The Second Quarterly NERVA Radiation Effects Program Meeting was attended at AGC/REON on January 10-11, followed by an informal meeting between personnel from General Dynamics/Fort Worth and NBS (on January 12, in Boulder) to discuss details of the thermal conductivity program. Another meeting with GD/FW was held in Boulder on March 6-7 to familiarize General Dynamics project personnel with actual NBS thermal conductivity program experimental procedures and techniques, and to discuss further the GD/FW work. A meeting was held in Boulder on March 21-22 with representatives from SNPO-C, GD/FW, AGC/REON and NBS to freeze the radiation effects program thermal conductivity experimental apparatus design and to discuss the new para-to-ortho hydrogen conversion experiment. In addition to these meetings, numerous telephone conversations have been held with all participating organizations on this work. A "Summary of Consultation Activity" covering a year and one half of NBS participation in this area is now being written and will be submitted to SNPO-C in early April.

On January 23-25, detailed discussions were held in Boulder with Dr. Landon Nichols, the NASA (SNPO) Contract R-45 technical monitor, on the hydrogen properties TAB Code, and current and forthcoming material properties work in the areas of thermal conductivity, electrical conductivity, thermal expansivity and thermometry.

3.3 Hydrogen Contamination - R. O. Voth, D. B. Chelton, and R. W. Arnett

3.3.1 Project Objectives

The objectives of the program are to determine the hazard associated with the contamination of liquid hydrogen with solid air and to develop means for minimizing the hazard and/or safely disposing of the contaminated mixture.

3.3.2 Project Status

The project is in the preliminary planning stage. Several important variables must be determined before design of the experimental apparatus can be initiated. A literature search has been completed and some preliminary tests are planned to supply needed information.

A preliminary test is planned to measure the settling time for solid nitrogen and solid oxygen particles in liquid hydrogen. The test will be run in a strip-silvered glass Dewar using a light source diametrically opposite a light meter which will measure the transmitted light. The data obtained from this test will be used in the design of the final cryostat.

4. Cryogenic Flow Processes

4.0 General Comments

Personnel contributing to the project during the present reporting period were J. A. Brennan, W. G. Steward, and P. F. Dickson.

4.1 Analysis of Transfer Line Cooldown

In this reporting period the analytical work was devoted to a treatment of the relatively long gradual cooldown period following the initial rapid transients. Since the calculation of these transients required extremely small time steps in the numerical integration, the computation time per unit of actual flow time was prohibitive for the complete cooldown. The approach to overcoming this difficulty has been to damp out the fluctuations so that changes are not so rapid and longer time steps become permissible. This shift to quasi-steady flow was accomplished by changing over to a steady flow continuity equation once the significant information about surges had been computed.

The use of longer time steps, however, created new problems and necessitated a more accurate integration method which consumed more computation time, partially nullifying the advantages. Despite these difficulties the computation time per unit of flow time has been improved by a factor of twenty. Sufficient flow time has been calculated to reveal that the wall temperature distribution does not yet agree well with experiments indicating a need to further refine the procedure.

4.2 Experimental Transfer Line Cooldown

The 1/4-in. O.D. transfer line has been installed and some preliminary tests completed. No results are presented since the tests completed were in the nature of shake-down runs.

Testing will proceed during the next reporting period on both liquid hydrogen and liquid nitrogen.

4.3 Heat Transfer in the Critical Region

A survey of theoretical and experimental work available on heat transfer in the region near the critical point is being conducted. Correlation of heat transfer results in this region is extremely difficult due to rapid variation of physical properties with temperature.

Scope of this study is:

Phase 1) assemble the available pertinent data,

Phase 2) tabulate most useful heat transfer correlations presently available for regions removed from the critical,

Phase 3) compile the unusual effects that occur in the critical region and explain their cause.

Phase 4) In light of this knowledge, develop from theoretical principles a useful correlation-prediction technique for heat transfer in the critical region.

At present, a literature search has been largely completed (Phase 1) as well as tabulation of correlations applicable to regions removed from the critical (Phase 2). Investigation and explanation of unusual effects in the critical region is in progress.

5. Cryogenic Propellant Venting Under Low Pressure Conditions

5.0 General Comments

Personnel contributing during this period were M. C. Jones and P. J. Giarratano.

5.1 Accomplishments During Current Reporting Period and Status of Project

Modifications were made to the test apparatus to insure safe operation with liquid hydrogen as the test fluid. Modifications included:

1. enclosing liquid hydrogen precooler with vent to outside of test laboratory;
2. plumbing for pressurizing liquid hydrogen storage dewar (850-liter dewar);
3. repair of discharge chamber to preclude possibility of liquid air collecting in chamber;
4. enclosing all electronic and power equipment in nitrogen-purged cabinets.

Preliminary runs have been completed and heat transfer measurements will commence during the next reporting period.